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**ADVANCED THERMALLY STABLE JET FUELS
DEVELOPMENT PROGRAM ANNUAL REPORT,
VOLUME III FUEL LUBRICITY**

**Southwest Research Institute
6220 Culebra Road
San Antonio, Texas 78228**

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<p>A program to evaluate the effects of additives on JP-8 fuel lubricity at high temperatures is described. Operating temperatures to 163°C were examined, with a subsequent program goal of 315°C. Baseline data were obtained using the Ball-on-Cylinder Lubricity Evaluator (BOCLE). High-temperature data were obtained with an apparatus known as the Cameron-Plint High Frequency Reciprocating Machine. The most significant effect of variables observed was a consequence of a change in wear specimen material from 52100 steel to M-50 steel--the latter considered to be representative of advanced fuel system designs. Use of M-50 wear coupons resulted in considerably reduced wear levels with only slight separation between neat or additized fuels. M-50 specimen roughness was a major influence in wear level. At finalized test conditions, a dibasic acid ester, di(2-ethylhexyl) adipate, was found to give the lowest wear values when used as an additive in clay-treated JP-8, although distinctions between the various additized fuels were not large. This finding is considered encouraging since the diester should be thermally stable at temperatures</p>					
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approaching the ultimate program goal of 315°C. The diester could also prove to be attractive as an alternative to the current JP-7 fuel lubricity additive--following more in-depth study of any deleterious effect on other fuel properties.

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FOREWORD

The program reported herein was conducted by the Fuels and Lubricants Research Division of Southwest Research Institute (SwRI), San Antonio, TX, under contract to Sandia National Laboratories, Albuquerque, NM. This work was supported by the U.S. Department of Energy at Sandia National Laboratories under Contract DE-AC04-76DP00789. Funding was provided by the Fuels Branch of the Air Force Aero Propulsion and Power Laboratory, Wright Research and Development Center (WRDC), Wright-Patterson AFB, OH. J.P. Cuellar, Jr. of SwRI served as the Principal Investigator. Mr. E.A. Klavetter was the Sandia Project Monitor, and Mr. W.E. Harrison, III of WRDC was the overall Program Manager. Messrs. Michael Baird and Swenam Lee, Pittsburg Energy Technology Center, were the DOE Program Managers.

The author acknowledges the technical contribution of Dr. C.A. Moses of SwRI. A major portion of the experiments was performed by J.J. Dozier in the laboratories of the U.S. Army's Belvoir Fuels and Lubricants Research Facility located at SwRI. The author also acknowledges the Belvoir Research, Development and Engineering Center, Materials, Fuels and Lubricants Laboratory, Fort Belvoir, Virginia, which has the managerial responsibility for, and permitted use of, the Belvoir Fuels and Lubricants Research Facility (SwRI).

This report is the third volume of a set of reports that documents work performed to develop advanced jet fuels that are thermally stable and have acceptable lubricity properties at high temperatures.

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SECTION I

INTRODUCTION

In recent years, the increasing utilization, unavoidably, of poor quality crude oils has necessitated the application of more severe refinery processing, especially hydrotreating, for fuel production. This processing serves to remove or convert trace polar compounds, heteroatom compounds, etc., to produce high quality fuels. Unfortunately, these compounds also impart a measure of inherent lubricating capability in a fuel which provides protection of mechanical systems under boundary lubrication operating conditions.

For U.S. Military aircraft turbine fuels, reduced lubricity as a consequence of this processing is mitigated by the requirement for a corrosion inhibitor/lubricity improver additive qualified to Military Specification MIL-I-25017. While the efficacy of these additives is generally accepted, it has also been recognized that differing degrees of effectiveness could be expected between the various qualified materials. In order to quantify additive effectiveness, a test method for the measurement of fuel lubricity was needed. This requirement was recently satisfied by the finalization of a standard apparatus and procedure¹ for a device known as the Ball-on-Cylinder Lubricity Evaluator (BOCLE). Although original versions of the BOCLE have been employed for over 20 years, development of a repeatable test apparatus and technique intensified in recent years, largely as a consequence of Air Force guidance/sponsorship.

The primary perceived deficiency of the BOCLE as a predictor of fuel suitability for aircraft systems is that it has no provision for operation at fuel temperatures much higher than ambient (the standard test procedure controls the test fuel at 25°C). Since current engine systems frequently operate at fuel temperatures of 105°C, with some as high as 135°C, it would not be surprising to find that the BOCLE ranks the various lubricity-improver additives differently than actual aircraft systems. Moreover, it is anticipated that advanced engine concepts under consideration will result in fuel temperatures reaching 163°C, with potential increases to 315°C. At these temperature levels, it is conceivable that an entirely different chemical class of additives will be required for lubricity enhancement.

The possibility of using the aircraft fuel in some airframe control systems is also contemplated in certain advanced applications. As a consequence, wear characteristics of a MIL-L-7808 engine lubricant and a MIL-I-83282 hydraulic fluid were also briefly examined in this study.

¹American Society for Testing and Materials Method D5001-89, Standard Test Method for Measurement of Lubricity of Aviation Turbine Fuels by the Ball-on-Cylinder Lubricity Evaluator (BOCLE).

SECTION II

OBJECTIVES AND APPROACH

The objectives of the investigation reported here were (1) to develop/modify a fuel lubricity test apparatus capable of high-temperature operation, and (2) to evaluate the effectiveness of current and candidate lubricity-improver additives at such temperatures (initial target--163°C, ultimate target--315°C). Throughout the additive evaluation, baseline data were obtained with the BOCLE at standard test conditions for a comparison of results.

Candidate lubricity additives for the high-temperature environment were solicited by correspondence with 15 major additive formulators. Recommendations were also requested of researchers at the Air Force Materials Laboratory and SwRI personnel with experience in this area.

For high-temperature lubricity evaluations, a modified version of a device known as the Cameron-Plint High-Frequency Reciprocating Machine was used in the investigation. The machine is capable of test temperatures in excess of 315°C. However, above the initial target temperature of 163°C, it will be necessary to pressurize the fluid system to avoid fuel boiling.

SECTION III

APPARATUS AND TEST FLUIDS/ADDITIVES

A. BALL-ON-CYLINDER LUBRICITY EVALUATOR

The BOCLE apparatus and operating procedures are described in detail by ASTM Method D 5001. Standard test conditions, unless otherwise noted, were used for all determinations performed. Major parameters were:

Test duration	30 min.
Load	9.8 N (1 kg)
Cylinder (ring) speed	240 rpm
Fuel temperature	25°C
Fuel volume	50 mL

B. CAMERON-PLINT MACHINE

The Cameron-Plint machine is a friction and wear device incorporating reciprocating motion with capability for operations at high temperatures (up to 600°C); it was this unique temperature capability which made the machine attractive for this investigation. The apparatus is illustrated in Figure 1. The contact configuration was ball-on-flat, with the ball receiving a reciprocated motion in a horizontal plane, with a pure sinusoidal motion. The scotch yoke which provides this motion was driven by a variable speed motor set at 20 Hz. The length of the stroke was 15 mm. The fixed, flat specimen was held in a stainless steel bath mounted upon a heater block. The bath was carried on flexures to permit measurement of the friction force. The load was applied directly to the reciprocating ball specimen by a spring balance. The stationary specimen carrier was electrically insulated, and the electrical contact resistance between the specimen pair was sensed by a Lunn-Furey potential divider circuit to provide an indication of film formation between the specimens.

In this study, the load was varied over a range of 10 to 40 N. The flat specimen temperature was controlled at either 30° or 163°C. The test specimens used in the Plint machine consisted of a 6.4-mm bearing ball loaded against a fixed rectangular flat specimen. The test balls were grade 10, AISI 52100 steel or, in the later stages of the study, M-50 steel. The flat specimens were, initially, of polished (3 in. centerline average) T-15 tool steel or M-50 steel. Surface roughness for the latter was a subject of investigation, variable from about 2 to 28 in. CLA. The specimens were immersed in the fuel to a depth of 2.5 mm. Test fluid flow through the bath was controlled at 10 mL/min. In some instances, an inert (nitrogen) atmosphere was maintained in the specimen/fluid area by enclosing the area with a Lucite cover.

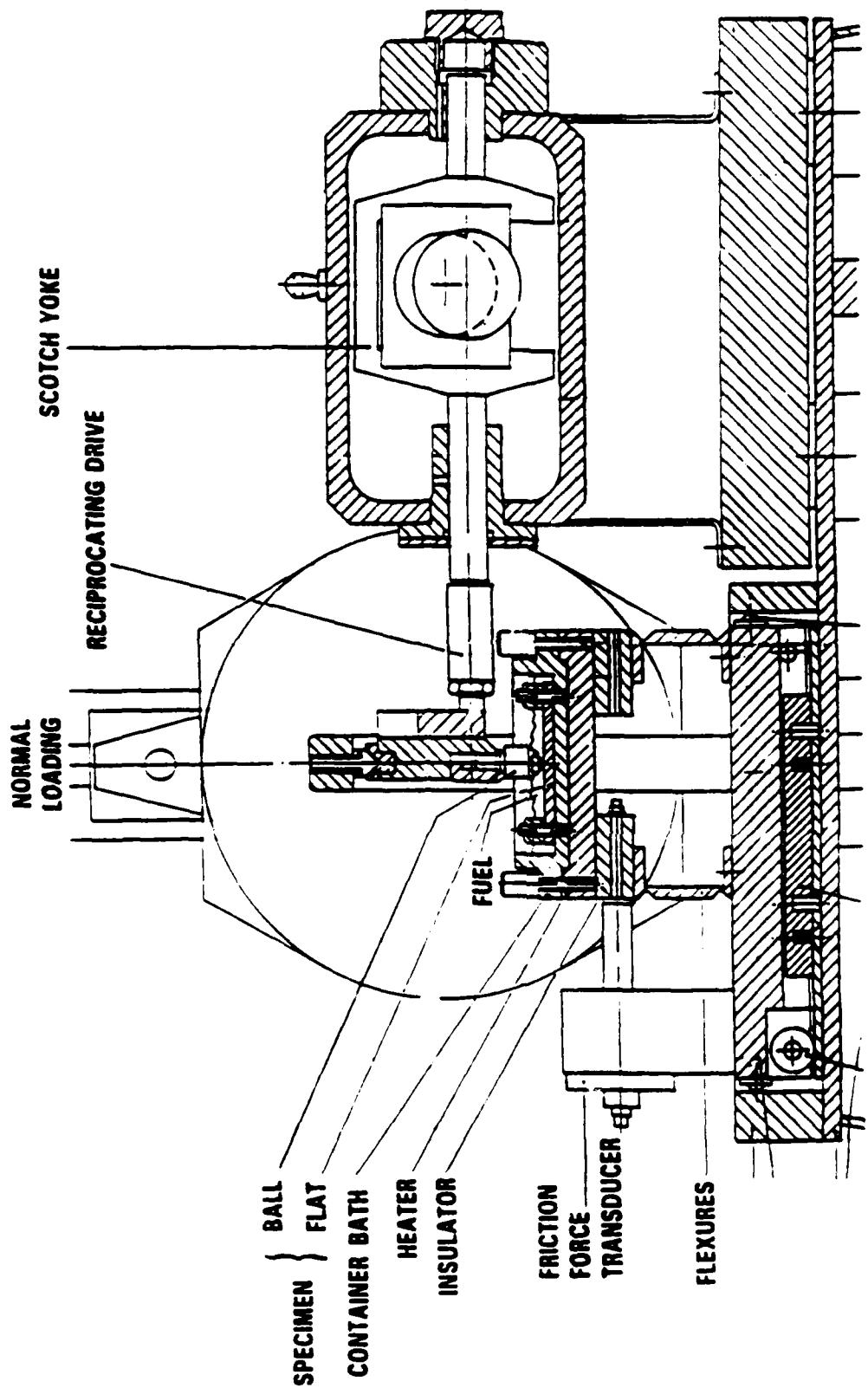


FIGURE 1. SCHEMATIC OF CAMERON-PLINT MACHINE

Wear data for the Plint machine are expressed in millimeters, to the nearest 0.01 mm, of the diameter of the circular wear scar produced on the test ball. The measurement was taken by means of an optical microscope with graduated reticle at 100X magnification.

C. TEST FLUIDS/ADDITIVES

Two "fuels" served as the primary test fluids for this investigation. These were an Air Force-supplied JP-8 engine fuel, and ISOPAR M, an isoparaffinic solvent which serves as the BOCLE reference fluid. In most cases, the JP-8 was clay treated to provide a low lubricity fluid to evaluate the efficacy of additives. The treatment procedure was that prescribed in ASTM Method D 2550 for the preparation of a "reference fluid base"--except that the volume of clay was twice that required by the method.

Since, in its present configuration, the Cameron-Plint machine does not incorporate a pressurized test section, the initial boiling point of the test fuels was of concern with respect to the maximum target temperature of 163°C. An IBP of 173°C was determined for the JP-8, while ISOPAR M has a typical value of 207°C. Although these values are below the maximum test temperature of 163°C, this would not preclude the possibility of discrete fuel vaporization associated with localized heating in the specimen contact zone.

Other test fluids which were evaluated for typical wear characteristics included a JP-4 and a JP-5 engine fuel, a MIL-L-7808 engine lubricant, and a MIL-I-83282 hydraulic fluid. These materials were all provided by the Air Force.

To avoid the appearance of endorsement, all commercially-supplied additives have been coded by letter. Additives A through E are currently approved to MIL-I-25017 as lubricity improver materials for use in JP-4, JP-5, and JP-8 fuel. Additives F through O were experimental type materials provided by various companies as possible high-temperature lubricity improvers. PWA-536 is the lubricity additive specified by MIL-T-38219 for use in JP-7 fuel.

Five unconventional type materials, in the sense that they are not considered normal fuel additives, were also selected for cursory evaluation. These were tricresyl phosphate (TCP), which is an antiwear agent frequently used in turbine engine lubricants; zinc dialkyldithiophosphate (ZDDP), an antiwear additive used in many automotive crankcase lubricants; two synthetic ester lubricant basestock materials, di(2-ethylhexyl) adipate (DEHA) and trimethylolpropane triheptanoate (TMP); and a polymer ester trade name product (additive P). The ester materials were included because they would be expected to impart some lubricity improvement at high temperatures and exhibit good thermal stability at such temperatures. It was conjectured that the TCP and ZDDP additives could also prove to be effective lubricity improvers at high fuel temperatures, but a question remains as to whether phosphorous-containing compounds might produce deleterious combustion products which would attack downstream engine components.

SECTION IV

EXPERIMENTAL RESULTS AND DISCUSSION

A. BOCLE WEAR TESTS

Baseline wear data using the BOCLE at standard test conditions were obtained for several qualified aircraft fluids (fuels and lubricants). Table 1 presents results obtained for three typical turbine fuels, a MIL-L-7808 engine lubricant, and a synthetic hydrocarbon, MIL-H-83282 hydraulic fluid. As expected, the latter two products showed significantly smaller wear scar diameters (WSD) relative to the three fuels. Data for the fuels indicated a measurable spread in lubricity performance between the fluids. However, the results should not be used to rank the fuel specifications since no information is available as to the history of the fuels or the type or concentration of lubricity-improver additives present.

**TABLE 1. BOCLE WEAR TEST RESULTS
FOR TYPICAL AIRCRAFT FLUIDS**

Test Fluid	BOCLE WSD, mm
JP-4 turbine fuel	0.58
JP-5 turbine fuel	0.50
JP-8 turbine fuel	0.54
MIL-L-7808 engine lubricant	0.44
MIL-H-83282 hydraulic fluid	0.39

Table 2 lists BOCLE results for some approved MIL-I-25017 additives (additives A through E) and the JP-7 lubricity additive (FWA-536), plus ten experimental type materials (additives F through O) which were submitted by various suppliers. In requesting samples for evaluation, the requirement for use at high temperature (163°C and, ultimately, 315°C) was emphasized. In some cases, suppliers recommended the additive currently qualified to MIL-I-25017. In general, the additive concentration used in the evaluation was that proposed by the supplier. Concentrations of 9 and 22.5 g/m³ correspond to the minimum effective concentration (MEC) and maximum allowable concentration (MAC) of most additives on the qualified products list for MIL-I-25017. A value of 200 g/m³ for PWA-536 is approximately equivalent to the JP-7 fuel specification requirement of 250 ppm MAC.

TABLE 2. BOCLE WEAR TEST RESULTS FOR SUBMITTED ADDITIVES

Additive	Conc, g/m ³	BOCLE WSD, mm
ISOPAR M Base Fuel		
None	--	0.84
A	9	0.64
B	9	0.65
C	9	0.58
D	9	0.63
E	9	0.64
E	22.5	0.55
L	9	0.46
L	22.5	0.48
PWA-536	200	0.74
Clay-Treated JP-8 Base Fuel		
None	--	0.80
A	9	0.60
A	22.5	0.54
B	9	0.66
B	22.5	0.54
C	9	0.58
C	22.5	0.59
E	9	0.68
E	22.5	0.62
PWA-536	200	0.72
F	22.5	0.60
G	22.5	0.59
H	22.5	0.80
I	22.5	0.80
J	22.5	0.80
K	9	0.68
L	9	0.50
M	9	0.59
N	9	0.78
O	9	0.81

The derived experimental repeatability of the BOCLE is a function of the wear level, i.e.,

$$\text{repeatability} = 0.109 (\text{WSD})^{1.8}$$

At the MIL-I-25017 specification limit of 0.65 mm, the repeatability value of 0.05 applies as the maximum difference between two test results, using the same apparatus and operation, in 95 percent of cases.

As seen in Table 2, all of the MIL-I-25017 materials met the specification criterion of 0.65 mm maximum wear scar in ISOPAR M at the MEC. Within this group, additive C showed the least wear. Experimental additive L, as a consequence of its performance in clay-treated JP-8, was run in ISOPAR M and indicated significantly reduced wear at 9 g/m³--notably superior to any of the MIL-I-25017 additives. At the higher concentration of 22.5 g/m³, additive L showed no further wear improvement. Results for the JP-7 lubricity improver, PWA-536, at its MAC showed the material to be the least effective of those evaluated in ISOPAR M.

Table 2 data using clay-treated JP-8 as the base fuel indicated no significant change in wear, versus ISOPAR M results, for the MIL-I-25017 additives or the JP-7 additive. All of the experimental additives (F through O) were examined in JP-8, and most were found to be comparable to or less effective than the MIL-I-25017 materials. Additive L, however, was the exception in that it showed the least wear by an appreciable margin.

BOCLE wear test results for five materials which are atypical of conventional fuel lubricity additives are given in Table 3. The two lubricant type antiwear agents, TCP and ZDDP, were examined up to relatively high concentrations since their use in lubricants can be as high as about 2 percent. Both materials were most effective at concentrations above or near 200 g/m³ in ISOPAK M. At 5000 g/m³, the zinc additive resulted in no wear. This may have been a consequence of a hydrodynamic film formation since the ZDDP product is quite viscous. All of the "additives" were evaluated at 200 g/m³ to obtain a comparison with the JP-7 lubricity agent and, in ISOPAR M, all showed equivalent or improved performance over the JP-7 additive. In general, however, the unconventional materials indicated greater effectiveness in the isoparaffinic solvent than in clay-treated JP-8. This was especially evident with ZDDP.

With regard to the foregoing, it is emphasized that BOCLE results, obtained at the standard fuel temperature of 25°C, do not relate to the program objective of high-temperature performance evaluation. Thus, additive performance changes or reversals at 163°C, or the ultimate test temperature of 315°C, would not be unexpected.

**TABLE 3. BOCLE WEAR TEST RESULTS
FOR UNCONVENTIONAL ADDITIVES**

		Wear Scar Diameter, mm	
Additive*	Conc, g/m ³	ISOPAR M	C-T JP-8
TCP	0	0.84	0.80
	9	0.76	--
	200	0.70	0.78
	500	0.50	--
	5000	0.40	--
ZDDP	0	0.84	0.80
	9	0.92	--
	200	0.52	0.86
	500	0.39	--
	5000	0.00	--
DEHA	0	0.84	0.80
	200	0.74	0.76
TMP	0	0.84	0.80
	9	0.78	--
	200	0.60	0.72
P	0	0.84	0.80
	200	0.62	0.66

*TCP--tricresyl phosphate

ZDDP--zinc dialkyldithiophosphate

DEHA--di(2-ethylhexyl) adipate

TMP--trimethylolpropane triheptanoate

P--additive code

B. CAMERON-PLINT WEAR TESTS

1. General

While some test conditions were varied within each category, the subsequent discussion is divided according to wear-specimen material type. Initial work employed available test specimens which had been used in other studies. These were a T-15 flat and 52100 ball--the same ball material used with the BOCLE. In anticipation of the ultimate goal of assessing lubricity at temperatures up to 315°C, experiments were performed using M-50 steel specimens. M-50 was selected to replace the 52100 steel since the latter shows significant loss of hardness at temperatures above 260°C; and one turbine engine fuel pump manufacturer advised that M-50 is more typical of advanced current and future engine designs. Work with different wear test

materials was conducted with M-50 balls, which were readily available, and the T-15 flat specimens. In the final task, the T-15 flat was also replaced by M-50.

2. Results With 52100 or M-50 Ball and T-15 Flat

A Plint machine test series to determine the effect of test time and, to some extent, test repeatability was conducted with 52100 balls. Figure 2 presents these results obtained for neat ISOPAR M and additized JP-8 (additive A at its MAC). The data indicate reasonable repeatability and show that the major portion of wear occurred within the first half-hour of run time. This is the expected outcome since specimen wear causes the load to be spread over a larger surface area with increasing time. As a consequence, most Plint tests were run for a 30-minute duration.

The effects of temperature (30° or 163°C) and ball material (52100 or M-50) for three additized fuels are given in Table 4. The last column in this table provides an indication of the extent of film formation in terms of the maximum reading of contact resistance during the test. A 100-percent reading would represent virtually complete separation between the ball and flat wear specimens. A "maximum" value for film formation is listed because in many instances the phenomenon was variable and transient--some tests indicated a film only in the early minutes of the run, some only in the later stages of the run, and some throughout the test. The results indicate, invariably, some film formation with the M-50 specimens and considerably reduced wear versus 52100 data. In general, the magnitude of the film was not an indication of wear, but did correlate with an observed reduction in friction force.

The results of Table 4 do not indicate a clear-cut effect for temperature. This may be attributable to competing mechanisms involved in the wear process. Increasing test temperature would be expected to exert a deleterious effect on wear. However, with an oxidizing (air) atmosphere at elevated temperature, degradation of the test fuel could result in products which improve fluid lubricity. The exclusion of oxygen by means of an inert atmosphere affects a third mechanism in the wear process, i.e., corrosive wear, whereby metal oxides are readily formed and worn away. It is generally held² that one of the major functions of current MIL-I-25017 lubricity additives is preferential chemisorption of the additive at the metal surface to the exclusion of oxygen and, therefore, mitigation of the corrosive wear process.

Table 5 gives the results of a brief series of tests using clay-treated JP-8 and an inert (nitrogen) atmosphere with the Plint machine. In general, inerting resulted in some wear reduction, with the most pronounced effect seen for the additized fuels with the 52100 ball and 163°C test temperature. Figure 3 better illustrates the effect of variables with the JP-8 fuel. Clearly the most predominant effect was exhibited by the M-50 ball material in the reduction of wear--essentially overshadowing all other variables. The cause of the apparent reversal of the effect of nitrogen on the nonadditive fuel at 163°C using 52100 is conjectural. With no additive present, the effect of fuel oxidation was, presumably, more predominant.

²Grabel, L., "Lubricity Properties of High Temperature Jet Fuel," Naval Air Propulsion Test Center, NAPTC-PE-112, August 1977.

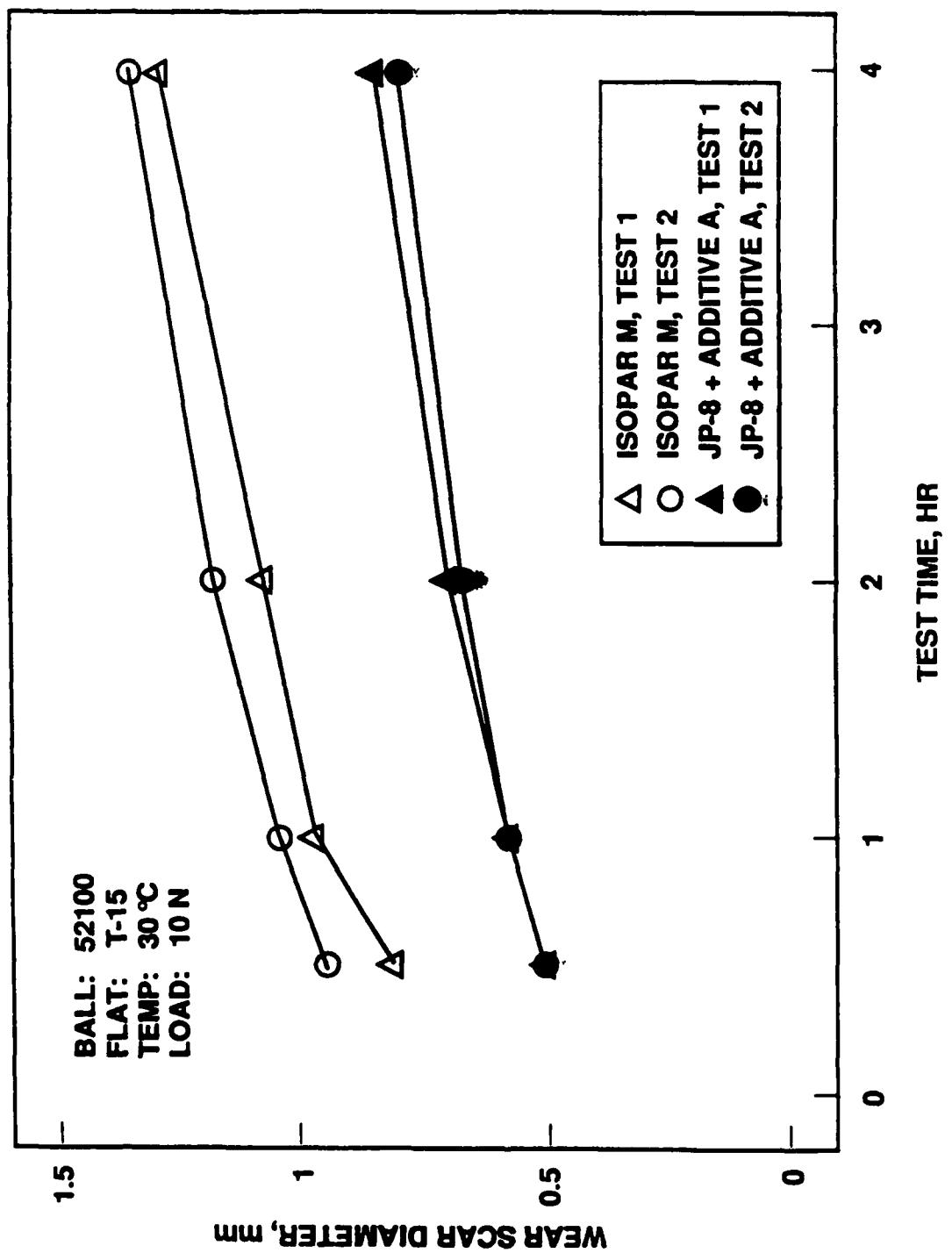


FIGURE 2. EFFECT OF TIME ON CAMERON-PLINT WEAR RESULTS

**TABLE 4. CAMERON-PLINT RESULTS FOR
CONVENTIONAL ADDITIVES
(T-15 flat; 10 N load; 30-min test)**

Additive at MAC	Temp, °C	Ball	WSD, mm	Film, % of scale
ISOPAR M Base Fuel				
None	30	52100	0.84	None
	163	52100	0.71	None
	30	M-50	0.48	4
	163	M-50	0.28	2
A	30	52100	0.53	None
	163	52100	0.69	None
	30	M-50	0.25	38
	163	M-50	0.28	1
B	30	52100	0.51	None
	163	52100	0.69	None
	30	M-50	0.25	40
	163	M-50	0.25	4
Clay-Treated JP-8 Base Fuel				
None	30	52100	0.69	None
	163	52100	0.69	None
	30	M-50	0.36	12
	163	M-50	0.30	5
A	30	52100	0.48	None
	163	52100	0.69	None
	30	M-50	0.25	30
	163	M-50	0.28	13
B	30	52100	0.51	None
	163	52100	0.69	None
	30	M-50	0.25	36
	163	M-50	0.28	1
PWA-536	30	52100	0.76	None
	70	52100	0.61	None
	115	52100	0.58	None
	163	52100	0.41	89
	30	M-50	0.23	88
	70	M-50	0.25	91
	115	M-50	0.25	84
	163	M-50	0.23	98

**TABLE 5. CAMERON-PLINT RESULTS WITH CLAY-TREATED JP-8
AND INERT ATMOSPHERE
(T-15 flat; 10 N load; 30-min test)**

Additive at MAC	Temp, °C	Ball	WSD, mm	Film, % of scale
None	30	52100	0.66	None
	163	52100	0.76	None
	30	M-50	0.30	30
	163	M-50	0.25	4
A	30	52100	0.43, 0.46*	None, None*
	163	52100	0.51, 0.51*	None, None*
	30	M-50	0.23, 0.20*	46, 52*
	163	M-50	0.25, 0.23*	2, 4*
B	30	52100	0.43	None
	163	52100	0.53	None
	30	M-50	0.25	50
	163	M-50	0.25	2

* Test fuel nitrogen blown.

The overriding influence of the M-50 material was also shown by results with the BOCLE, as follows:

<u>Ball Material</u>	<u>Wear Scar Dia, mm</u>
<u>Clay-treated JP-8</u>	
52100	0.86
M-50	0.46
<u>C-T JP-8 + Additive A, MAC</u>	
52100	0.54
M-50	0.44

Wear reduction using the M-50 ball and clay-treated fuel was greater than that shown for the additive using the 52100 ball. With M-50, wear with or without the additive at its maximum allowable concentration was essentially equivalent.

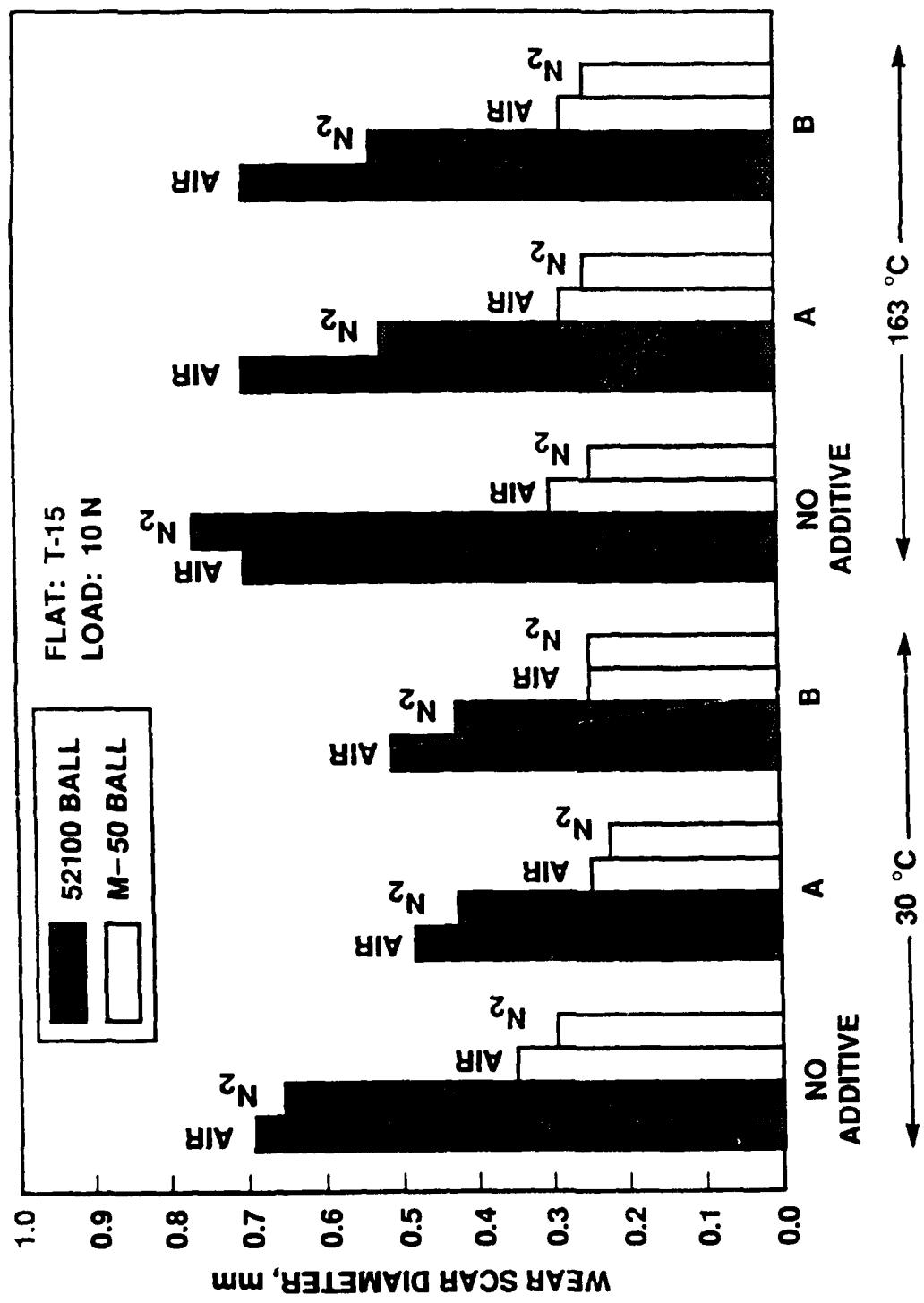


FIGURE 3. EFFECT OF VARIABLES ON CAMERON-PLINT WEAR RESULTS
 WITH CLAY-TREATED JP-8 BASE FUEL

Cameron-Plint test results for the 7808 engine lubricant and the 83282 hydraulic fluid are shown in Table 6. Unexpectedly, the engine lubricant displayed relatively poor wear performance, especially at the higher test temperature. The hydraulic fluid also indicated a deleterious temperature effect with the 52100 ball, but not with M-50.

3. Results With M-50 Ball and M-50 Flat

To match the M-50 ball, M-50 flat specimens were commercially procured. Subsequent experiments with these specimens were found to be nonrepeatable. The problem was traced to surface roughness discontinuities in the flat specimens and, as a consequence, a number of determinations were invalidated. To recover the specimens, they were machine polished to a roughness equivalent to the T-15 coupons previously used, i.e., 3 in. CLA (centerline average).

Initial tests with these reworked specimens showed mild wear with little separation between the clay-treated JP-8 and additized JP-8 (additive A at 22.5 g/m³). To determine whether improved separation between these fuels could be achieved at higher loads, two test series were conducted with increasing load at 30-minute intervals, and at a 30°C test temperature. The following data were obtained:

Load, N	Wear Scar, mm	
	C-T JP-8	JP-8 + Additive A
0	0.28	0.20
20	0.30	0.23
30	0.33	0.25
40	0.38	0.28
50	0.41	0.30

Although the above results displayed consistently increasing wear versus load for both series, the separation between the clay-treated base fuel and the additized fuel remained relatively slight.

In an effort to increase the degree of wear and, possibly, the separation between fuels, the remaining M-50 flat specimens were reworked by machine grinding to a higher surface roughness of approximately 16 in. CLA. Results with these specimens at 30 °C were obtained in a load range of 20 to 40 N, shown here:

Load, N	Wear Scar, mm	
	C-T JP-8	JP-8 + Additive A
20	0.54	0.42
30	0.53	0.43
40	0.65	0.46

**TABLE 6. CAMERON-PLINT RESULTS FOR AIRFRAME FLUIDS
(T-15 flat; 10 N load; 30-min test)**

Fluid	Temp, °C	Ball	WSD, mm	Film, % of scale
MIL-L-7808J	30	52100	0.64	6 None
	163	52100	0.81	
	30	M-50	0.23	98
	163	M-50	0.56	None
MIL-H-83282C	30	52100	0.28	98
	163	52100	0.48	96
	30	M-50	0.23	89
	163	M-50	0.23	95

The greater roughness did serve to increase wear and to some extent separation between fuels. The effect of load on specimen wear was somewhat mitigated.

At this point in the investigation, it became necessary to fix upon a "standard" set of test conditions for the evaluation of candidate additives. Since the use of polished flat specimens appeared to be unrealistic with respect to both aircraft fuel system simulation and wear levels, it was decided to approximate a specimen roughness equivalent to an actual main engine, gear type, fuel pump. Consequently, measurements were made of the gear tooth surface for a J57 engine fuel pump. Values of about 25 in. CLA were obtained in both the axial and perpendicular directions for an available unused pump. Accordingly, M-50 Plint specimens were ground to that approximate roughness.

A specimen load of 20 N was selected to provide a reasonable wear level without excessive surface stress. In addition, it was decided to emphasize use of an inert test atmosphere (although some comparative experiments were also performed in air) to avoid the complication of fuel oxidation and its effect on lubricity. Moreover, it is believed that the limited availability of oxygen, other than fuel-dissolved, is more representative of an aircraft fuel system which is relatively closed.

A summary of the finalized conditions for additive evaluation is shown as follows:

Test duration	30 min
Load	20 N
Reciprocating velocity	20 Hz
Stroke	15 mm
Ball	M-50, Grade 10
Flat	M-50, 26 in. CLA
Fuel flow	3 mL/min
Temperature	30°/163°C
Atmosphere	N ₂ /Air

The results of experiments at the above conditions are given in Table 7. The program schedule did not permit development of statistically significant repeatability data for the test at finalized conditions. As indicators of experimental error, the average wear scar differences between duplicate runs were calculated. These values were 0.04 mm with air and 0.03 mm with a nitrogen atmosphere. Some few runs were performed at 30°C in nitrogen to obtain an indication of the effect of temperature (30° vs 163°F). At 30°C, additive A and the DEHA diester provided slight wear reduction. The 83282 hydraulic fluid showed a similar effect, while the 7808 engine lubricant was equivalent to the clay-treated JP-8.

The 163°C test results given in Table 7 indicated somewhat increased wear as a consequence of the higher temperature. Of the qualified MIL-I-25017 additives evaluated (additives A through E), A and B were somewhat more effective. Although some of the submitted candidate additives were roughly comparable to A and B, none showed a significant improvement. In particular, additive L, which indicated superior performance in standard BOCLE testing, did not exhibit any exceptional beneficial effect using the Plint machine with finalized conditions. In contrast, the JP-7 fuel additive, PWA-536, indicated relatively high wear values in the BOCLE, but was among the better materials listed in Table 7. The antiwear agent, TCP, showed comparable performance to PWA-536, while the polyol ester, TMP, gave a relatively high wear value of 0.82 mm.

At a concentration of 22.5 g/m³, the dibasic acid ester, DEHA, provided little or no wear improvement. However, at 2000 g/m³, the ester gave the lowest wear value of the materials investigated. Although other materials, e.g., additives A and B, were comparable considering the experimental error, the DEHA result is particularly encouraging, since the fluid would be expected to remain thermally stable at even higher temperatures. Moreover, it is possible that effectiveness at some intermediate concentration to those examined might be obtained which would permit DEHA to be used as an economically attractive replacement for the current JP-7 fuel lubricity additive, PWA-536. Of course, such use would have to be examined relative to any effect the tester might have on other fuel properties. One of the more critical of these properties is fuel thermal stability as measured by the Jet Fuel Thermal Oxidation Tester according to ASTM Method D 3241. A preliminary determination of this property was made using the JP-8 base fuel with 2000 g/m³ DEHA. At the fuel specification requirement of 260°C tube temperature, the additized fuel met all performance criteria.

Wear results in Table 7 for the two airframe fluids at 163°C indicate both materials were no better than the clay-treated JP-8 at these conditions. This finding is unexpected since moderately high viscosity, fully-formulated lubricants/hydraulic fluids of this type would be conjectured to provide better wear protection.

Some comparative tests with an oxidizing (air) atmosphere at 30°C are also given in Table 7. In comparison with the 30°C data using nitrogen, the oxidizing condition resulted in slightly higher wear levels.

**TABLE 7. ADDITIVE EVALUATION WITH THE CAMERON-PLINT
MACHINE AT FINALIZED CONDITIONS
(Clay-Treated JP-8 Base Fuel)**

Additive			
Code	Conc, g/m ³	Test Temp, °C	*Wear Scar Dia, mm
N₂ Atmosphere			
None	--	30	0.66
A	22.5	30	0.58
DEHA	2000	30	0.61
7808	lubricant fluid	30	0.66
83282		30	0.60
None	--	163	0.75
A	22.5	163	0.68
B	22.5	163	0.68
C	22.5	163	0.72
D	22.5	163	0.74
E	22.5	163	0.80
F	22.5	163	0.70
G	22.5	163	0.75
I	22.5	163	0.85
L	22.5	163	0.72
P	2000	163	0.75
PWA-536	200	163	0.70
TCP	2000	163	0.70
TMP	2000	163	0.82
DEHA	22.5	163	0.76
DEHA	2000	163	0.66
7808	lubricant fluid	163	0.77
83282		163	0.77
Air Atmosphere			
None	--	30	0.76
A	22.5	30	0.69
B	22.5	30	0.65
C	22.5	30	0.70
D	22.5	30	0.69
E	22.5	30	0.69
L	22.5	30	0.70
7808	lubricant fluid	30	0.64
83282		30	0.65

* Average of duplicate determinations.

All of the experiments listed in Table 7 exhibited some film formation as evidenced by the Plint machine contact resistance measurements. These values were in the range of 1 to 11 percent of full-scale for all fluids except the MIL-H-83282 fluid which consistently showed values of 75 to 85 percent.

With regard to the foregoing discussion of wear results using the M-50/M-50 specimen pair, it is emphasized that performance differences between test fuels/fluids were subtle. The predominant effect was that shown by specimen roughness, with a marked increase in wear level as a consequence of increased roughness.

SECTION IV

CONCLUSIONS

The following conclusions are drawn from the investigation reported herein:

- Initial screening of candidate lubricity additives using the Ball-on-Cylinder Lubricity Evaluator at standard conditions identified only one material (additive L) which was significantly superior within the group of conventional type additives.
- The program objective to develop/modify a fuel lubricity test apparatus capable of high-temperature operation was met through utilization of the Cameron-Plint High Frequency Reciprocating Machine. There were no operational problems encountered with the machine at temperatures up to the initial target of 163°C. The apparatus is capable of much higher test temperatures if a pressurized fuel system can be successfully incorporated.
- In early test series to examine the effects of temperature, inert atmosphere, additives, etc., the most predominant effect, by far, was that shown by the change from 52100 steel to M-50 steel wear specimens. While the use of M-50 type steel is considered a requirement to simulate the high-temperature fuel systems of advanced aircraft, Cameron-Plint test results with M-50 resulted in only slight differences in wear performance between fluids. Wear level with M-50 specimens was significantly affected by surface roughness. From the foregoing, it may be concluded that the longest wear life for contacting components in a fuel system can be achieved by using M-50 type steel in fabrication and the smoothest surface roughness which is economically feasible.
- In the final evaluation of additives at "standard" conditions, a dibasic acid ester, di(2-ethylhexyl) adipate, was found to result in the lowest wear when used as an additive in clay-treated JP-8. Since the diester would be expected to exhibit reasonable thermal stability at even higher temperatures, the significance of this finding is two-fold. First, use of this fluid as a lubricity additive up to 315°C, the ultimate program target, should be possible. Second, with further study, the diester could prove to be an economically attractive replacement for the current JP-7 lubricity additive. Since the use of diesters as fuel additives is believed to be a unique application not previously explored, a patent disclosure for the application has been prepared.

SECTION V

RECOMMENDATIONS

The obvious recommendation is that the fuel lubricity phase of the overall program on advanced fuels should be extended to include the final goal of fuel temperatures up to 315°C. This subsequent step will necessitate modification of the Cameron-Plint machine to provide pressurization of the test section and fuel delivery system.

In a separate objective, it is felt that further investigation is warranted to determine whether diesters might serve as economical lubricity additives in JP-7 fuel. This work should include the effect of molecule chain length, optimum concentration, and any effects on fuel specification properties.